UNITED STATES DEPARTMENT OF AGRICULTURE

AGRICULTURAL MARKETING SERVICE

DAIRY DIVISION GRADING AND INSPECTION LABORATORY

1819 W. Pershing Road
Chicago 9, Illinois

METHODS OF LABORATORY ANALYSIS

FOR

EVAPORATED MILK

AND

CANNED WHOLE MILK

SEPTEMBER 1955

Agriculture - Washington

For Administrative Use
Dairy Division Laboratory

Determination of Net Weight

The odd-numbered cans of evaporated milk or canned whole milk are weighed to an accuracy of 0.01 ounce. These cans are opened, emptied into clean, dry containers and contents poured back and forth until mixed uniformly. The cans are washed clean, dried, cooled to room temperature and weighed. Net weight is calculated. If average weight of the odd-numbered cans is less than 14.5 ounces (or weight stated on label) the even-numbered cans are also weighed and the average net weight of both odd and even-numbered cans calculated.

Determination of Solids

From the (above mentioned) well-mixed sample of evaporated milk weigh approximately 1.0 gram (analytical balance) into a Mojonnier type solids dish which has been previously cleaned, dried, heated on 180°C. hot plate, cooled to room temperature and weighed. Add 1 ml. distilled water, mix with rotary motion until sample is completely incorporated with water. Place dish on 180°C. hot plate and uniformly evaporate moisture to light tan shade. Transfer dish to the vacuum oven at 100°C. for 10 minutes at 25 inches or more vacuum. Then transfer dish to desiccator and cool to room temperature (in Mojonnier about 7 minutes) and weigh. Calculate percentage of total solids. If the analysis shows total solids to be below specification the test is repeated in duplicate on each even-numbered can in that composite.

The total solids test for canned whole milk is the same as for evaporated milk with two exceptions, (1) Approximately 2 grams of sample are weighed into the solids dish and (2) No distilled water is added to solids dish.

Determination of Fat

Approximately 5.0 grams of well-mixed sample of evaporated milk are weighed on analytical balance into Mojonnier fat flask or are weighed with weighing

pipette and transferred to the fat flask. Five ml. of distilled water are added and mixed. For canned whole milk weigh approximately 10 grams of sample and do not add the distilled water. Next add 1.5 ml. of 28 percent ammonium hydroxide and mix. Add 10 ml. ethyl alcohol and mix. A drop or two of 1 percent phenolphathalein is optional. Add 25 ml. ethyl ether, stopper and mix vigorously. Add 25 ml. petroleum ether, stopper and again mix vigorously. Place flasks in Mojonnier centrifuge and centrifuge 60 revolutions in 1 minute. Stratification may be obtained also by holding flasks - usually about 20 minutes. Decant ether layer into Mojonnier type fat dish which has been cleaned, dried, heated on 135°C. hot plate, cooled to room temperature and weighed. Evaporate ether slowly. Make a second extraction by adding 5 ml. ethyl alcohol, 25 ml. ethyl ether and 25 ml. petroleum, otherwise proceed the same as for the first extraction.

For the third extraction omit the alcohol and proceed same as for the second extraction. If needed, pour distilled water down side of flask to raise aqueous layer level to center of the contricted section of fat flask before last extraction. Pour third ether layer into fat dish as completely as possible without including any of the aqueous layer. After the ether has evaporated from dish, place dish in vacuum oven for 5 minutes at 135°F. and 20 inches or more vacuum. Transfer dish to desiccator and cool to room temperature. Weigh and calculate percentage of fat.

If the analysis for fat is below specifications the test is repeated in duplicate on each even-numbered can in the composite.

Reference: (1) "Official Methods of Analysis of the Association of Official Agriculture Chemists", 7th Edition, page 249, para. 15.74 Association of Official Agricultural Chemists, P.O. Box 540, Benjamin Franklin Station, Washington 4, D. C.

Determination of Flavor

Evaporated milk or canned whole milk is tempered to 80 to 90°F. and observed for odors and flavors other than the normal clean, sweet and characteristic heated flavor.

Determination of Body

In making the observations on body the cans should not be shaken before opening. It is preferable to hold the sample cans overnight, then open. Observe for lack of uniformity and separation of fat or protein as contents are slowly poured from can into a glass container.

Determination of Color

Observe sample in adequate daylight or under ample whitelight fluorescent light for abnormal color, lack of uniform color and for shades that are darker than a light cream color, that was caused by excessive heating or by storage.

Determination of Sediment

Weigh 225 grams of evaporated milk into 1500 ml. beaker, add sufficient hot, sediment free water to facilitate filtering. Filter through 1-1/8" diameter area of a standard lintine sediment pad and rinse with sediment free water. Compare visually with USDA sediment standards for milk and milk products. Measure one pint (473 ml.) of canned whole milk and proceed in same manner as described above.

Examine nature of the material under a wide field microscope and compare amount of material with United States Department of Agriculture Sediment Standard for Milk and Milk Products.

Determination of pH

Electrometric method of pH determination is recommended. Proceed according to manufacturer's instructions for the particular instrument used.

Standard Plate Count

One end of the can is washed with soap and water. Sponge with 5 percent phenol or submerge it for 2 minutes in solution with 200 p.p.m. of available chlorine. Shake can to mix contents well. Dip surface to be punctured in alcohol and flame just long enough to dry. Can is opened aseptically.

For the standard plate count one ml. of milk or 0.5 ml. of evaporated milk is inoculated into 4 ounce prescription bottles with 25 ml. of tempered (45°C) sterile agar. Mix well and cool in a horizontal position. Duplicate bottles are incubated at 32°C. and duplicates are incubated at 55°C. Higher inoculations are advocated if colonies can be visually identified in the agar. The agar is tha t specified in the latest edition of Standard Methods for the Examination of Dairy Products.

Reference: "Standard Methods for the Examination of Dairy Products." Latest Edition.

For prescence of anaerobic organisms inoculate in duplicate 1 ml. of canned milk or evaporated milk into bottom of a tube with 10 ml. of thioglycollate medium. Seal with approximately one-half inch of sterile paraffin. Incubate at 32°C. for 72 hours and examine tubes for bacterial growth and gas. A set of controls is prepared in each testing period.

The thioglycollate media are prepared by rehydrating and sterilizing fluid thioglycollate (dehydrated) according to the manufacturer's direction.